JOINT INVENTORS

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APPLICATION FOR UNITED STATES LETTERS PATENT

SPECIFICATION

TO ALL WHOM IT MAY CONCERN:

Be it known that we, Elisha Stav, a citizen of Israel, residing at 28 Woodcrest Drive, Amherst 14226, in the State of New York, Edward A. Burkard, a citizen of The United States of America, residing at 8395 Stahley Road, East Amherst 14051, in the State of New York, and Ronald S. Finkelstein, a citizen of The United States of America, residing at 241 Wellingwood Drive, East Amherst 14051, in the State of New York, have invented a new and useful "Cementitious Gypsum-Containing Compositions and Materials Made Therefrom" of which the following is a specification.

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CEMENTITIOUS GYPSUM-CONTAINING COMPOSITIONS AND MATERIALS MADE THEREFROM

CROSS REFERENCE TO RELATED APPLICATIONS

This is a continuation-in-part of ~~pending~~ U.S. application Serial No. 08/253,333 filed June 3, 1994, also abandoned

BACKGROUND OF THE INVENTION

Field of the Invention

The invention relates to cementitious compositions and in particular to cementitious construction materials such as floor underlayments, backer boards, floor and road patching materials, fiberboard, fire-proofing sprays, and fire-stopping materials made from a composition comprising gypsum, Portland cement and silica fume.

Description of Related Technology

Construction materials, such as backer boards for showers and floor underlayments, typically do not contain gypsum because gypsum-containing materials are usually not water resistant. However, gypsum is a desirable component in construction materials due to its rapid cure and early strength characteristics. Attempts to improve the water-resistance of gypsum boards by mixing Portland cement and gypsum (calcium sulfate hemihydrate) have met with limited success because such a mixture can result in the formation of ettringite, which causes expansion of the gypsum/Portland cement product and thus leads to its deterioration. Ettringites are formed when tricalcium aluminate ($3CaO \cdot Al_2O_3$) in the Portland cement reacts with sulfate.

A cementitious composition useful as a pavement patching compound which contains Portland cement and alpha gypsum is disclosed in Harris, U.S. Patent No. 4,494,990. The composition also includes a pozzolan source, such as, for example, silica fume, fly ash or blast furnace slag. The Harris patent discloses that the pozzolan blocks the interaction between the

tricalcium aluminate and the sulfate from gypsum. The Harris patent discloses mixing a three-component blend of Type I Portland cement, alpha gypsum and silica fume with a fine aggregate to prepare a mortar used to cast mortar cubes for evaluating the strength of the resulting composition.

Ortega et al., U.S. Patent No. 4,661,159 discloses a floor underlayment composition that includes alpha gypsum, beta gypsum, fly ash and Portland cement. The patent also discloses that the floor underlayment material can be used with water and sand or other aggregate to produce a fluid mixture which may be applied to a substrate.

SUMMARY OF THE INVENTION

It is an object of the invention to overcome one or more of the problems described above.

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~~According to the invention, a cementitious composition includes about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate, about 10 wt.% to about 40 wt.% Portland cement, about 4 wt.% to about 20 wt.% silica fume, and about 1 wt.% to about 40 wt.% pozzolanic filler. The invention further includes construction compositions and materials made from the inventive cementitious composition and methods for making the same.~~

Other objects and advantages of the invention will be apparent to those skilled in the art from the following detailed description taken in conjunction with the drawings and the appended claims.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a cross-sectional view of a covered board according to the invention.

Fig. 2 is a graph depicting compressive strength vs. curing time for a composition #1 according to the invention and a comparative composition #2.

Fig. 3 is a scanning electron microscope (SEM) micrograph (500x) of a board made from a composition according to the invention disclosed in Example 3.

Fig. 4 is an SEM micrograph (100x) of the board shown in Fig. 3.

Fig. 5 is an SEM micrograph (1000x) of the board shown in Fig. 3.

DETAILED DESCRIPTION OF THE INVENTION

According to the invention, a composition for use in construction materials is provided which is particularly useful in areas where water resistance is an important consideration, such as for backer boards for baths and showers, floor underlay applications and exterior sheathing boards. Further uses of the inventive composition include materials such as self-leveling floors and road patching materials, fire-proofing sprays, fire-stopping materials, and fiberboard.

~~Compositions according to the invention~~ include about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate (i.e., beta-gypsum), about 10 wt.% to about 40 wt.% Portland cement (Type III is preferred), about 4 wt.% to about 20 wt.% silica fume, and about 1 wt.% to about 40 wt.% pozzolanic filler.

The beta-gypsum component of the inventive composition is calcium sulfate beta hemihydrate, commonly referred to as stucco. Beta-gypsum is traditionally less expensive than alpha-gypsum ~~and is typically not as strong because it comprises very small crystals of hemihydrate held together in porous conglomerates, while alpha-gypsum comprises well crystallized prisms of hemihydrate.~~ However, boards made from the inventive composition have exhibited more than adequate strength for interior applications such as backer boards and floor underlayments and exterior applications, such as exterior sheeting and eaves.

The Portland cement component of the composition according to the invention is preferably Type III Portland cement (according to ASTM standards). Type III Portland cement cures faster than Type I and Type II Portland cement and exhibits an early high strength.

The silica fume component of the composition according to the invention is an extremely active pozzolan and prevents the formation of ettringite.

The pozzolanic filler component of the composition according to the invention may be a natural or man-made filler that contains a high percentage of amorphous silica. Natural pozzolanic fillers are of volcanic origin and include trass, pumice, and perlite. Man-made pozzolanic fillers include fly ash and Fillite (Fillite Division of Boliden Intertrade, Inc. Atlanta, Georgia).

Pozzolanic fillers contain a high percentage of amorphous silica which possesses little or no cementitious properties. However, in the presence of moisture, pozzolanic fillers have surfaces that are chemically reactive with calcium hydroxide at standard temperatures to form hydrated calcium silicate (CSH) which, in compositions and methods according to the invention, are believed to become a homogeneous part of a cementitious system due also to the presence of the finely divided pozzolan of the invention, such as silica fume. Compositions according to the invention which include both a pozzolanic filler and finely divided pozzolan result in cementitious materials wherein the transition zone between the filler and a cement paste is densified and thus produces a cured product of higher compressive strength than compositions which utilize a pozzolanic filler alone or a finely divided pozzolan alone. It is believed that the mechanism which causes changes in the microstructure of compositions according to the invention to result in higher compressive strengths is associated with two effects: a pozzolanic effect and a micro-filler effect (due to the fine size and spherical shape of the silica fume).

Compositions for construction materials such as backer boards and floor underlays according to the invention preferably include about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate (about 30 wt.% to about 50 wt.% is preferred), about 10 wt.% to about 40 wt.% Portland cement (about 6 wt.% to about 25 wt.% is

preferred), about 4 wt.% to about 20 wt.% silica fume (about 4 wt.% to about 10 wt.% is preferred), and about 10 wt.% to about 40 wt.% a pozzolanic filler (about 25 wt.% to about 35 wt.% is preferred). A preferred filler for use in such construction materials is pumice. Pumice is desirable as it is relatively light weight and can be sized to result in a product of desirable strength and physical properties. For example, Hess Pumice Products Inc. manufactures a size No. 10 pumice aggregate that measures about 93% greater than 1400 microns, while the size No. 5 pumice aggregate has a particle size measurement of about 23% greater than 1400 microns. Although fillers such as calcium carbonate, crystalline silica and different types of clay could be included in the composition, it has been found that the use of a pozzolanic filler results in a product according to the invention having superior properties. As explained above, this is believed to occur because the surfaces of the pozzolanic filler react with free lime to form hydrated calcium silicate (pozzolanic reaction) which becomes part of the product matrix. Such a reaction is only possible with pozzolanic fillers.

The composition according to the invention produces building materials which set up quickly, exhibit high strength and durability, and are water resistant. Furthermore, traditional gypsum board producing machinery may be utilized to produce a board from a composition according to the invention without modification of the machinery. Because the composition according to the invention sets up quickly (typically in three minutes or less), building materials made from the composition can be handled (e.g. sheets can be cut into smaller sheets or boards) much faster than products made from Portland cement alone. Furthermore, unlike traditional gypsum board, boards or other products made from a composition according to the invention do not require kiln drying, and in fact, kiln ~~drying should be avoided.~~

With reference to Figure 1, a backer board 1 according to the invention comprises a core 3 made from

a cementitious composition according to the invention and adjacent cover sheets 5 and 7 disposed at either side thereof. Such a board may be manufactured by the following process:

Raw gypsum may be calcined at about 160°C (320°F) to about 175°C (374°F) to form calcium sulfate hemihydrate. The calcined gypsum can be post-ground to a finer particle size if, for example, certain strengths, water requirements, and working properties are desired. The gypsum powder is fed to a mixer and blended with Portland cement, silica fume and a pozzolanic aggregate filler. The pozzolanic filler may be pumice, perlite, trass, or fly ash or a mixture thereof. Other ingredients that may be included in the composition are set control additives (e.g. accelerators), water reducing agents, water repellent additives and latex or polymer modifiers. The resulting blend is combined with a slight stoichiometric excess of water to produce a slurry. The slurry, which forms the core 3 of the board, is poured onto a lower, continuous cover sheet 5 which is disposed on a conveyor. Then, an upper continuous cover sheet 7 is placed on the core as it moves on the conveyor. The cover sheets 5 and 7 are preferably made from fiberglass matt, fiberglass scrim, or a composite of both. The cover sheets may also be made from polyethylene, polypropylene or nylon; however, such materials are not as desirable as fiberglass as they are more expensive. As the slurry sets, ~~dihydrate needles form and interlock with the cover sheets~~. As the covered board moves along the conveyor line in a continuous sheet, the board gains sufficient strength so that it can be handled. The board is then cut into sections, (for backer boards, usually either 3 ft. x 5 ft. or 3 ft. x 4 ft. sheets) and transferred to pallets. The board thickness preferably ranges between about 1/8 inch and about 5/8 inch. The boards are then preferably stacked and cured from one to seven days (particularly preferred about three days) at a temperature of about 16°C (60°F) to about 27°C (80°F) (i.e. room temperature) and a humidity of about 40% to about 70%, after which the

boards may be sent to a customer. The stacking of the boards advantageously provides a moist environment for curing. The boards may be cured at temperatures and humidities outside of the above-stated ranges resulting ~~in an acceptable product. However, this may extend the~~ curing time. A board according to the invention usually reaches its full strength about fourteen to about twenty-eight days after formation.

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When preparing a board or other product according to the invention, the forced drying required for gypsum board should be avoided. An alternative curing procedure is to wrap the boards in plastic wrapping for about three days to retain moisture for continuous curing. Such wrapped boards have exhibited about 50% higher strength than normal gypsum boards of the same density. Also, the wrapped boards develop about 70% to about 80% of their ultimate strength in three days.

When a board or other product having a thickness of about 1/8 inch is desired, the cementitious composition thereof preferably includes about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate, about 10 wt.% to about 40 wt.% Portland cement, about 4 wt.% to about 20 wt.% silica fume, and about 1 wt.% to about 40 wt.% pozzolanic filler, resulting in a very strong thin product, especially useful, for example, for floor underlayments. A preferred cementitious composition for use in very thin boards (i.e. about 1/8 inch) and floor underlayments includes about 70 wt.% to about 75 wt.% calcium sulfate beta hemihydrate (about 74 wt.% is particularly preferred), about 15 wt.% to about 25 wt.% Portland cement (about 20 wt.% is particularly preferred), about 4 wt.% to about 8 wt.% silica fume (about 6 wt.% is particularly preferred), and about 1 wt.% to about 10 wt.% pozzolanic filler.

Compositions according to the invention may also be used to prepare self-leveling floor compositions and road patching materials. In such materials, a master blend composition according to the invention is prepared which includes about 30 wt.% to

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about 75 wt.% calcium sulfate beta-hemihydrate (i.e. beta-gypsum) (about 30 wt.% to about 50 wt.% is preferred), about 10 wt.% to about 40 wt.% Portland cement (about 6 wt.% to about 25 wt.% is preferred), about 4 wt.% to about 20 wt.% silica fume (about 4 wt.% to about 8 wt.% is preferred), and about 1 wt.% to about 40 wt.% a pozzolanic filler (about 1 wt.% to about 15 wt.% is preferred; about 1 wt.% to about 5 wt.% particularly preferred). The master blend is then mixed with silica aggregates (i.e., predominately quartz local sand) to form the floor or road patching material.

Preferably, a self-leveling floor composition according to the invention includes (i) about 25 wt.% to about 75 wt.% of the master blend; and (ii) about 75 wt.% to about 25 wt.% sand. Most preferably, a self-leveling floor composition master blend includes about 71 wt.% calcium sulfate beta-hemihydrate, about 20 wt.% Portland cement, about 6 wt.% silica fume and about 2 wt.% Fillite pozzolanic filler. Because of its low density, Fillite addition of amounts as low as about 1 wt.% of the composition provide a considerable volume of filler (see Example 2, Table II for Fillite physical properties).

A road patching composition according to the invention includes (i) about 25 wt.% to about 100 wt.% of the master blend described herein with respect to the self-leveling floor compositions of the invention; and (ii) about 75 wt.% to about 0 wt.% sand.

Compositions according to the invention may also be used in fiberboards according to the invention. Such fiberboards include (i) about 70 wt.% to about 90 wt.% of the master blend described herein with respect to the self-leveling floor compositions and road patching compositions of the invention; and (ii) about 30 wt.% to about 10 wt.% of a fiber component. The fiber component is preferably selected from the following: wood fibers, paper fibers, glass fibers, polyethylene fibers, polypropylene fibers, nylon fibers, and other plastic fibers.

Most preferably, a master blend according to the invention for use in such a fiberboard includes about 74 wt.% calcium sulfate beta-hemihydrate, about 20 wt.% Portland cement, and about 6 wt.% silica fume.

Fire-proofing sprays and fire-stopping materials may also be prepared utilizing compositions according to the invention. Such fire-proofing and fire-stopping materials include about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate (about 30 wt.% to about 50 wt.% is preferred), about 10 wt.% to about 40 wt.% Portland cement (about 6 wt.% to about 25 wt.% is preferred), about 4 wt.% to about 20 wt.% silica fume (about 4 wt.% to about 10 wt.% is preferred), and about 1 wt.% to about 40 wt.% a pozzolanic filler (about 1 wt.% to about 10 wt.% is preferred). Preferably, the pozzolanic filler is Fillite or perlite or mixtures thereof. Fire-proofing sprays and fire-stopping materials according to the invention also preferably include about 1 wt.% to about 30 wt.% unexpanded vermiculite filler. Such fire-proofing and fire-stopping materials may also include up to about 2 wt.% glass fibers and up to about 2 wt.% of a thickening agent. The thickening agent is preferably selected from the following: cellulose derivatives, ~~acrylic resins and mixtures thereof.~~

EXAMPLE 1

A cementitious composition according to the invention was prepared with components set forth in the amounts stated in Table I below:

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TABLE I

Material	Weight Percent
Beta-gypsum (Stucco)	45.1
Type III Portland Cement	19.2
Silica Fume	9.5
Pumice Filler	24.6
Perlite	1.47
W.R.A.[1]	0.87
Water Repellent Agent[2]	0.11
Accelerator (ball-milled $CaSO_4 \cdot 2H_2O$ gypsum dihydrate[3])	0.042

[1] Water reducing agent or wetting agent including lignosulfonates and/or naphthalene sulfonates manufactured by Georgia Pacific Corp. and Henkel Corp., respectively.

[2] A silicone product or like material, e.g., Veoceal 2100 and Veoceal 1311 (both TM designations of products manufactured by Wacker Silicone Corp.)

[3] See U.S. Patent Nos. 3,920,465, 3,870,538 and 4,019,920

The materials identified in Table I were mixed and 100 grams thereof was mixed with 35.6 grams of water. About 1 wt.% to about 5 wt.% of a polymer latex (acrylic or SBR) was added to the mixture to improve flexibility. The mixture was then formed into boards according to the invention using a glass matt/scrim composite. The boards were tested for water absorption, nail holding properties, deflection, compression strength (wet and dry), water wicking characteristics and other ASTM specification requirements. The boards met the ASTM specifications with respect to each test.

EXAMPLE 2

A self-leveling floor composition #1 according to the invention was prepared with the components set forth in the amounts stated in Table II below. A cementitious composition #2 with components also set forth in the amounts stated in Table II below

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(which did not include a pozzolanic filler) was also prepared.

TABLE II

Material	Composition #~~1~~2 (weight percent)	Composition #~~2~~1 (weight percent)
Beta-Gypsum (Stucco)	36.1	40.0
Type III Portland Cement	9.8	10.8
Silica Fume	2.96	3.24
~~Fillite~~ FILLITE 500 Pozzolanic Filler[1]	0.0	1.35
Sand (quartz; crystallized silica)	49.4	43.26
W.R.A.[2]	0.82	0.9
Retarder[3]	0.06	0.06
Anti-foaming agent[4]	0.33	0.26

[1] Fillite Division of Boliden Intertrade, Inc., Atlanta Georgia. Hollow silicate spheres with the following physical properties: average particle density of 0.6-0.8 g/cc; average bulk density of 0.35-0.45 g/cc; and typical particle size of 5-300 microns.

[2] Water reducing agent or wetting agent including lignosulfonates and/or naphthalene sulfonates manufactured by Georgia Pacific Corp. and Henkel Corp., respectively.

[3] A natural protein-based material.

[4] A vegetable oil-based dry powder.

In order to form a floor composition of a smooth consistency, composition #1 was mixed with about 26 wt.% water and composition #2 was mixed with about 24 wt.% water. The density of composition #1 was 107 lbs./ft^3. The density of composition #2 was 111.62 lbs./ft^3.

Both compositions were allowed to dry at about 21°C (70°F) ~~70°F~~ and a relative humidity of about 50%. The compressive strengths of samples (2 inch by 2 inch by 2 inch cubes) of each of the compositions were tested after 2 hours of drying, and after 1, 3, 7 and 28 days

by pressing in an Instron press according to ASTM C472-9A.

The results of the compressive strength tests are shown in Fig. 2. Composition #1 according to the invention exhibited a greater compressive strength than Composition #2 for all samples tested. Although the compressive strengths of both compositions were similar after curing for 28 days, the advantage of a composition according to the invention is evident when the densities of the two compositions are taken into consideration. Typically, a composition having a higher density should also exhibit a higher compressive strength. However, in this instance, Composition #1 according to the invention had a lower density than Composition #2, and yet exhibited a slightly higher compressive strength.

EXAMPLE 3

A cementitious composition according to the invention was prepared with components set forth in the amounts stated in Table III below:

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TABLE III

Material	Weight Percent
Beta-gypsum (Stucco)	35.9
Type III Portland Cement	15.6
Silica Fume	7.8
Pumice Filler	39.5
W.R.A.[1]	0.87
Water Repellent Agent[2]	0.11
Accelerator (ball-milled $CaSo_4 \cdot 2H_2O$ gypsum dihydrate[3])	0.058

[1] Water reducing agent or wetting agent including lignosulfonates and/or naphthalene sulfonates manufactured by Georgia Pacific Corp. and Henkel Corp., respectively.

[2] A silicone product or like material, e.g., Veoceal 2100 and Veoceal 1311 (both TM designations of products manufactured by Wacker Silicone Corp.)

[3] See U.S. Patent Nos. 3,920,465, 3,870,538 and 4,019,920

The materials identified in Table III were mixed and 100 grams thereof was mixed with 35.6 grams of water. About 1 wt.% to about 5 wt.% of a polymer latex (acrylic or SBR) was added to the mixture to improve flexibility. The mixture was then formed into boards according to the invention using a glass matt/scrim composite. The boards were tested for water absorption, nail holding properties, deflection, compression strength (wet and dry), water wicking characteristics and other ASTM specification requirements. The boards met the ASTM specifications with respect to each test.

The scanning electron microscope (SEM) micrographs shown in Figs. 3, 4, and 5 were made of a cured sample of Example 3. An arrow 30 points to pumice in the sample, illustrating that in a composition according to the invention, the pumice becomes part of the hydrated calcium silicate (CSH) matrix, substantially eliminating any transition zone 32 between the pumice filler and the cement paste.

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The foregoing detailed description is given for clearness of understanding only, and no unnecessary limitations should be understood therefrom, as modifications within the scope of the invention will be apparent to those skilled in the art.